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CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART IX GOLD, SILVER, PLATINUM, PALLADIUM AND THEIR ALLOYS

UDC 620·182·2:669·21/·23

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2

Price Rs 5·00 *June* 1976

Indian Standard

CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART IX GOLD, SILVER, PLATINUM, PALLADIUM AND THEIR ALLOYS

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Indian Standard

CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART IX GOLD, SILVER, PLATINUM, PALLADIUM AND THEIR ALLOYS

0. FOREWORD

0.1 This Indian Standard (Part IX) was adopted by the Indian Standards Institution on 31 December 1975, after the draft finalized by the Metallography and Heat-Treatment Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The primary object of metallographic examination is to reveal the constituents and the structure of metals and their alloys by means of a microscope. Because of diversity in available equipment, wide variety of problems encountered, and the personal element, this standard gives for the guidance of the metallographer only those practices which experience has shown are generally satisfactory.

0.3 This standard is being issued in parts. This part covers polishing, etching and examination of gold, silver, platinum, palladium and their alloys. The other parts of this code are as follows:

Part I	General features
Part II	Electrolytic polishing
Part III	Aluminium and its alloys and their examination
Part IV	Copper and its alloys and their examination
Part V	Iron and steel and their examination
Part VI	Lead and its alloys and their examination
Part VII	Magnesium and its alloys and their examination
Part VIII	Nickel and its alloys and their examination
Part X	Tin and its alloys and their examination
Part XI	Zinc and its alloys and their examination

1. SCOPE

1.1 This standard (Part IX) covers the polishing, etching and examination of gold, silver, platinum, palladium and their alloys.

2. PREPARATION OF SPECIMENS

2.0 Recommended methods of selection, size, cutting, cleaning and mounting of metallographic specimens are outlined in Part I of this standard.

2.1 Specimens of gold, silver, platinum, palladium and their alloys are all prepared in about the same way as the more common metals. A plane surface is first secured with a flat, single-cut file, after which the surface is ground successively on No. 240, 280, 320 and 400 grain Aloxite paper and No. 000 or 0000 metallographic emery paper (previously smoothed by rubbing with a bit of hardened steel). (For details of grit numbers of abrasive papers, *see* IS : 715-1966* and IS : 2832-1964†.) A comparative chart of grit numbers of abrasive grains is given in Appendix A.

2.2 The specimen is then polished on a felt wheel using No. 600 Alundum and kept well moistened. The final polish is secured with a velvet or velveteen wheel using finely divided magnesia (as a water paste) or levigated alumina (settled at the rate of 6 mm/h). When magnesia is used, the usual precautions to avoid carbonate formation shall be followed.

3. ETCHING REAGENTS

3.1 In Table 1 are given the etching reagents commonly recommended for the gold, silver, platinum, palladium and their alloys.

3.2 After etching, the specimen is immediately and thoroughly washed with distilled water, followed by rinsing in a suitable medium (alcohol, acetone, etc) and drying to avoid staining. The specimen should be preserved, if necessary, in a desiccator.

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*Specification for coated abrasives, glue bond (*second revision*).

†Specification for waterproof silicon carbide paper.

TABLE 1 ETCHING REAGENTS FOR GOLD, SILVER, PLATINUM, PALLADIUM AND THEIR ALLOYS

(*Clause* 3.1)

Sl No.	Etching Reagent	Composition*	Remarks	Use
(1)	(2)	(3)	(4)	(5)
i)	Potassium cyanide ammonium persulphate	a) KCN (5 percent solution) 1 part $(NH_4)_2S_2O_8$ (5 percent solution) 1 part	a) 1 to 2 minutes	a) Pure silver
		b) KCN (10 percent solution) 1 part $(NH_4)_2S_2O_8$ (10 percent solution) 1 part	b) Addition of 2 percent KI will produce more rapid attack. Etch for ½ to 3 minutes. Make up fresh each time. The etching rate may be increased by warming solution. KI additions may cause staining	b) Gold, nearly all carat golds
		c) KCN (20 percent solution) 1 part $(NH_4)_2S_2O_8$ (20 percent solution) 1 part	c) 3 percent KI may be added	c) A slow etch for palladium or the complicated dental alloys
ii)	Chromate	a) HNO_3 (1:1) 100 ml $K_2Cr_2O_7$ 2 g b) CrO_3 20 g Na_2SO_4 1·5 g H_2O 100 ml	Dilute (a) to 20 vol, add equal amount of (b). Apply with camel's hair brush. Non-adherent film of red silver chromate should form. If film adheres, add more of (a); if none forms, add (b)	Used for silver alloys

---

*The use of concentrated reagents is intended, unless otherwise specified.

(*Continued*)

TABLE 1 ETCHING REAGENTS FOR GOLD, SILVER, PLATINUM, PALLADIUM AND THEIR ALLOYS — *Contd*

SL No. (1)	ETCHING REAGENT (2)	COMPOSITION* (3)	REMARKS (4)	USE (5)
iii)	Chromic acid-sulphuric acid	CrO_3 (0·2 percent 1 part solution) H_2SO_4 (0·2 percent 1 part solution)	Etch for 1 minute	Silver alloys
iv)	Electrolytic etching	a) HF (1 percent solution with small amount of $SnCl_2$)	—	a) Used for silver-tin alloy with over 73 percent silver
		b) Dilute solution of HCl, KCN or KCN+KI	—	b) Ocassionally used for gold
		c) KCN (5 percent solution)	—	c) Used particularly for silver where silver is in contact with other metals as in plated materials
v)	Iodine	50 percent solution of I_2 in aqueous KI solution	Film of silver iodide may remain on surface of gold-silver alloys; can be removed with KCN solution	Gold alloys
vi)	Aqua regia	a) Slightly diluted	a) Etch for 5 minutes in warm solution	a) Pure platinum
		b) Concentrated	b) Use hot	b) Platinum alloys. Also used for gold alloys but will form chloride film when much silver is present. Ammonia or KCN will remove film
		c) In glycerin	c) Use cold	c) Palladium alloys

vii)	Potassium sulphide	K_2S		Use hot	Gold-nickel alloys
viii)	Fused salts	$KOH+10$ percent KNO_3 or $KHSO_4$		Etch in fused salt	Used for platinum if aqua regia is ineffective
ix)	Nitric acid	HNO_3		Use hot	Palladium
x)	Ammonium hydroxide-hydrogen peroxide	NH_4OH H_2O_2	5 parts 1-3 parts	Immersion	For silver or silver-palladium alloys and silver soldered joints
xi)	Dichromate etch	$K_2Cr_2O_7$ (saturated solution) NaCl (saturated solution) H_2SO_4	100 ml 2 ml 10 ml	Use 1 part solution and 9 parts water. Apply with swab	Silver and silver alloys
xii)	Ferric chloride	[Fecl$_3$ (Two percent aqueous solution)]		Etch for 5 to 30 s	Used for silver solders to show structural details

*The use of concentrated reagents is intended, unless otherwise specified.

APPENDIX A

(*Clause* 2.1)

COMPARATIVE CHART OF GRIT NUMBERS (APPROXIMATE) OF ABRASIVE GRAINS

Aluminium Oxide, Silicon Carbide and Garnet		Flint		Glass		Corundum		Emery		Trade Designation
IS Grit Number	B.S. Grade Number	IS Grit Number	B.S. Grade Number	IS Grit Number	B.S. Grade Number	IS Grit Number	B.S. Grade Number	IS Grit Number	B.S. Grade Number	
14	14	—	—	—	—	—	—	—	—	—
16	16	—	—	—	—	—	—	—	—	—
24	24	24	3	24	3	24	—	24	—	Extra Coarse
30	30	30	2½	30	2½	30	—	30	—	Extra Coarse
36	36	36	2	36	—	36	—	36	3	Coarse
40	46	40	1½	40	S2	40	—	40	2½	Coarse
50	54	50	1	50	M2	50	—	50	2	Medium Coarse
60	60	60	½	60	—	60	—	60	1½	Medium
80	80	80	—	80	—	80	—	80	1	Medium
100	100	100	0	100	F2	100	—	100	F	Medium Fine
120	120	120	00	120	1½	120	—	120	FF	Fine
150	150	150	—	150	1	150	—	150	—	Fine
180	180	180	—	180	0	180	—	180	0	Extra Fine
220	220	—	—	—	—	—	—	—	—	—

NOTE — Grits 240 and finer come under the sub-sieve range and as limits for these cannot be set on common silk test sieves, the grain sizes shall conform to general commercial grading, and it is recommended that the sedimentation process be adopted for their analysis.

INDIAN STANDARDS

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METALLOGRAPHY AND HEAT TREATMENT

IS:	
2756-1965	Method for estimating the average grain size of wrought copper and copper base alloys
2853-1964	Method for determining the austenitic grain size of steel
4075-1967	Methods of macro-streak flaw test for steel
4163-1967	Determination of inclusion content in steel by microscopic method
4748-1968	Method for estimating average grain size of metals
6396-1971	Methods of measuring decarburized depth of steel
6416-1971	Methods of measuring case depth of steel
7739 (Part III)-1975	Code of practice for preparation of metallographic specimens: Part III Aluminium and its alloys and their examination
7739 (Part IV)-1975	Code of practice for preparation of metallographic specimens: Part IV Copper and its alloys and their examination

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Printed at Britannia Calendar Mfg Co, Calcutta, India